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Comparative analysis of air dried alchornea laxiflora leaves (AALL) extracted with different solvents on corrosion inhibition efficiency of mild steel in acidic media

Oluwafemi L. Adebayo[1], Emmanuel F. Olasehinde[2], Labunmi Lajide[2], Daniel T. Oloruntoba[3]

[1]Visiting Scholar, Department of Chemical and Process Engineering, University of Strathclyde, Glasgow, United Kingdom
[2]Department of Chemistry, Federal University of Technology Akure, Ondo State, Nigeria.
[3]Department of Metallurgical and Materials Engineering, Federal University of Technology Akure, Ondo State, Nigeria.

Abstract

The objectives of this study were to determine the effects of four extractive solvents on the phytochemical activity of the air dried alchornea laxiflora leaves in corrosion prevention of mild steel in acidic medium. The dried plant samples were ground, separately sieved using 0.25µm and then extracted with ethanol, methanol, water and diethylether using maceration method. The percentage composition of each solvent extracts was determined and their phytochemical constituents were analyzed using appropriate methods. The phytochemicals detected were found to vary upon the solvent used for the extraction with methanol extract gave the highest content of the phytochemicals. Weight loss experiment was performed by varying the immersion time, concentration of extract and the temperature. It was found that the corrosion rate of the mild steel decreased with increase in concentration of the extracts but increases with increase in temperature while the inhibition efficiencies of the extracts increased with increase in concentration of the extracts but deceases with rise in temperature and immersion time, but a less pronounced effect was noticed with methanol extract. The maximum inhibition efficiency (%) obtained for the different solvents extracts at the highest concentration and lowest temperature studied are: methanol extract (89.39), ethanol extract (86.36), aqueous extract (81.75) and diethyl ether extract (64.29). The activation energy (EA), enthalpy change and entropy change which are the thermodynamic parameters were evaluated. Kinetics of the reaction in the presence of the extracts revealed that it follows a first order reaction and the half-life increase as the concentration of the extract increases.

Keywords: Alchornea laxiflora leaves, maceration, phytochemicals, inhibition efficiency, Mild steel

1. Introduction

Euphorbiaceae is the family name of Alchornea laxiflora plant and it is a deciduous shrub and about 6–10 m high. It grows naturally in Nigeria, in DR Congo, in Ethiopia, and throughout East Africa to Zimbabwe [1]. The plant is monoecious having its male and female inflorescences on separate branches. Alchornea laxiflora is called "Opoto" among the Yoruba tribe in Nigeria. The plant part serve many importance, for instance its leaves are used in preservation and in medicine as antimalaria [2] while the stem, especially the branches, is used in Nigeria as chewing sticks (local tooth brush) for cleaning teeth [3]. The plant is also useful among the Yoruba tribe of Southwestern Nigeria for the treatment of poliomyelitis and measles [4]. Apart from the antimicrobial effects of the plants on bacterial, its antioxidants properties have also been reported by some researchers [5]. The inhibitive properties of the plant leaves extract in corrosion prevention of Mild Steel in HCl have also been investigated. The results obtained from their investigation has revealed that the leaf extract of the alchornea laxiflora leaves contain alkaloids, flavonoids, saponins, tannins, carbohydrates, cardioactive glycosides steroids, phenols, and reducing sugars as phytochemicals resident in the plant [6].

Moreso, the antitoxicity, anticonvulsant, and sedative effects of the leaf extract of A. laxiflora in animal models have been reported by some researchers [7]. Meanwhile the effects of drying method on the plant leaves from our previous research have established that the air dried samples of the plant leaves extract have highest inhibition efficiency. The present study

Corresponding author: Adebayo Oluwafemi
Email Address: femtay41@yahoo.co.uk

focused on the inhibition study of the different solvents extract of the plant leaves on mild steel in 1.0 M HCl.

2. Materials and Methods

2.1 Materials

The mild steel employed for this study was procured and the chemical composition was determined at Federal University of Technology, Akure, Ondo State, Nigeria. The sheets were mechanically pressed cut to coupon of dimension $2.5 \times 2.5 \times 0.4$ cm. A small hole of about 5 mm diameter near the upper edge of the coupons was made to help hold them with glass hooks and suspended them into the corrosive medium. The mild steel was polished with different grades of emery paper, dried in acetone and stored in moisture free desiccators prior to use [8-10]. The aggressive acidic solution of 1.0 M HCl was prepared by dilution of concentrated HCl with distilled water and all experiment was carried out in unstirred solutions and all weighing was made using analytical weighing balance (Metler Toledo PB153). Other materials and equipment used include Alchornea laxiflora leaves, rotary evaporator, distilled water, beaker, measuring cylinder, thermostated water bath, thermometer, paper tape, whatman filter paper, desicator.

2.2 Preparation of plant sample

Alchornea laxiflora leaves were obtained in the vicinity of Federal Polytechnic Ado, Ekiti State, Nigeria and were authenticated at the Department of Biology, College of Education, Ikere, Ekiti State, Nigeria. The fresh samples were washed and air-dried for one week. All dried plant materials were ground to powder and sieved to obtain fine particles. 20 g of the sample was differently soaked with methanol, ethanol, diethylether and water with continuous agitation for 72 hours. The filtrates were concentrated using rotary evaporator at 60^0C. The dried extracts were weighed and stored at room temperature.

2.3 Determination of plant yield

The percentage yield was obtained by using equation 1,

$$\frac{W_2-W_1}{W_0} \times 100 \qquad (1)$$

Where W_2 is the weight of the extract and container, W_1 is the weight of the container alone and W_o is the weight of the original dried sample.

2.4 Determination of the phytochemical constituents

Biochemical test were done to check the presence and the amounts of different phytochemicals such as flavnoids, steroids, tannin, alkaloids, saponin, anthraquinone, cardiac glycosides, phytobalatin and terpenoid in the extract of the air dried alchornea laxiflora leaves and this was carried out by simple qualitative and quantitative methods [11-15].

2.5 Weight loss Measurement

In weight loss experiment, a previously weighted metal coupon was completely immersed in 100 mL of 1.0 M HCl in the absence and presence of different concentration (0.2g , 0.4g, 0.6g, 0.8g and 1.0g) of the solvents extracts with the aid of glass hooks at different temperature (303K, 313K,323K and 333K) for four hours. After every 4 hours, each coupon was withdrawn from the test solution, and the corrosion product was removed by washing each coupon in distilled water, rinsed in acetone and dried in the air completely before re-weighing. From the initial and final weights of the mild steel, the weight loss, corrosion rate (CR ,g hr^{-1} cm^{-2}) in absence and presence of inhibitors, the inhibition efficiency (I.E %) of the inhibitors and the degree of surface coverage were calculated using equations 2, 3 and 4 respectively [16].

$$CR = \frac{\Delta W}{At} \qquad (2)$$

$$I.E = 1 - \left(\frac{CR_2}{CR_1}\right) X\ 100 \qquad (3)$$

$$\text{Surface coverage } (\Theta) = 1 - \left(\frac{CR_2}{CR_1}\right) \qquad (4)$$

Where Δw is the weight loss in grammes, CR_1 and CR_2 are the corrosion rates of the mild steel strip coupons in absence and presence of inhibitor, A is the cross-sectional area in cm^2 and t is the exposure time in hours.

3. Results and Discussion

3.1 Effects of solvent on extraction yield

Table 1 showed the percentage yield of the different solvents extracts of air dried alchornea laxiflora leaf. It was revealed from the table that methanol gave the highest yield followed by ethanol, water and diethylether. This results showed that methanol is an efficient solvent in extracting phytochemicals from the air dried alchornea laxiflora leaves.

Table1: Percentage yield of air dried alchornea laxiflora leaves extracted with different solvents

Solvents	Percentage yield (%)
Ethanol	62.67
Methanol	78.23
H_20	58.54
Diethylether	50.12

3.2 Phytochemical screening

The phytochemical screening tests was performed to detect the presence of bioactive components and their quantity in all the different solvent extracts of air dried alchornea laxiflora leaves. The results obtained are presented in Table 2a and b.

Table 2a: Qualitative phytochemical screening of air dried alchornea laxiflora leave extracted with different solvents

Phytochemicals(mg/g)	ME	AE	EE	DE
Alkaloid	+	+	+	+
Flavnoid	+++	+	++	+
Anthraquinone	-	-	-	-
Tannin	+++	++	++	+
Terpenoid	++	+	+	+
Steroid	+	+	+	+
Saponin	+++	++	++	+
Cardiac glycoside	++	+	+	+
Phytobatanin	-	-	-	-

Table 2b: Quantitative phytochemical screening of air dried alchornea laxiflora leave extracted with different solvents

Phytochemicals(mg/g)	ME	AE	EE	DE
Alkaloid	7.91	5.43	3.63	2.46
Flavnoid	12.51	6.1	7.49	5.90
Tannin	16.07	-	10.58	7.10
Terpenoid	13.23	6.06	7.06	3.04
Steroid	5.36	4.89	4.37	3.59
Saponin	51.05	26.40	45.55	13.90
Cardiac glycoside	9.75	4.01	4.72	3.87

ME= methanolic extract, AE= Aqueous extract, EE= Ethanolic extract, DE= Diethylether extract

3.3 Effects of extracts concentration and Temperature on corrosion rates

The results of the corrosion rate of mild steel in 1.0 M HCl solution at different temperature in the absence and presence of different concentrations of the solvents extracts of air dried alchornea laxiflora leaves are shown in Figure1. The plots showed the corrosion rate versus extracts concentration of the different solvents extracts of air dried alchornea laxiflora leaves at the temperature range 303K -333K. The result revealed that as the concentration of the solvents extracts increases the corrosion rate of the mild steel in the corrosive media decreases. Meanwhile the rate of reduction in corrosion rate of the mild steel in the corrosive media is more pronounced with methanol extract. Moreso, careful examination of the effects of temperature on the rate of corrosion revealed that corrosion rate increased with increase in temperature of the acidic medium due to the desorption of the phytochemical constituents from the surface of the mild steel in the inhibited solution and the effects is much more pronounced on the surface of the mild steel in the uninhibited solution.

3.4 Effects of extract concentration and Temperature on Inhibition Efficiency

The effects of solvents extracts concentration on their inhibition efficiency at different temperature were also examined and the results are presented in Figure 2. From the results, it was revealed that the different solvent extracts protected the mild steel in acidic media. The protection of the mild steel in the acidic medium might be ascribed to the adsorption of the phytochemical constituents of the extracts on the surface of the mild steel and their inhibition efficiency increases with increase in concentration but decreases with increase in temperature as a result of increase in the average kinetic energy which led to the desorption of some of the adsorbed phytochemical constituents of the extracts on the surface of the mild steel. Similar findings have been reported by some researchers [16], [17]. Meanwhile comparative analysis of the inhibition efficiency of the ethanol, methanol, diethylether and aqueous extracts at the highest concentration and different temperature (Figure 3) studied, revealed that the methanol extract showed a better inhibitive effect on mild steel in acidic media due to its high extraction yield and phytochemical constituents which contains heteroatom's that serves as an adsorption sites of the extracts onto the mild steel surface.

3.5 Thermodynamic Studies

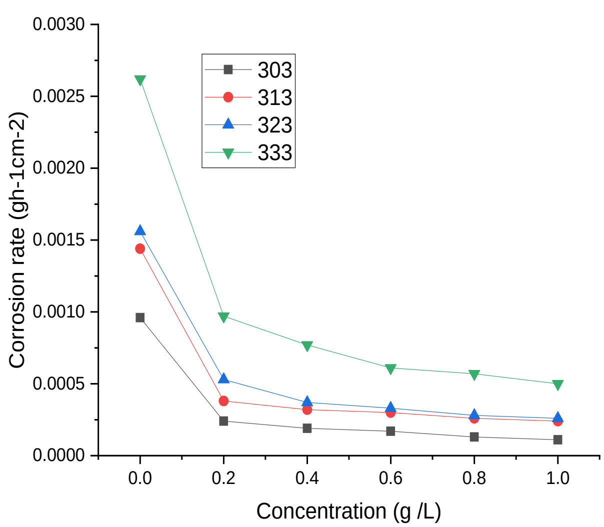
From the effects of the temperature variation on the corrosion rate of mild steel, the thermodynamic properties such as Activation Energy (Ea), Enthalpy (ΔH°) and Entropy (ΔS) change of activation are studied in order to identify the mechanism of adsorption process involved. The activation energy was evaluated from the effect of temperature on the corrosion rate of mild steel in 1.0 M HCl using Arhenius equation:

$$Log\ CR = -\frac{E_a}{2.303RT} + \log A \qquad (5)$$

Where CR is the corrosion rate, Ea is the activation energy, R is the gas constant and T is the temperature. A plot of log CR vs $\frac{1}{T}$ is a straight line graph from which the activation energy was obtained from the slope $(-\frac{E_a}{2.303RT})$ of the graph.The enthalpy change (ΔH°) and entropy (ΔS) change for all solvents extract were evaluated from the effect of temperature on the corrosion rate of mild steel in 1.0 M HCl using Errying Transition state equation.

$$log\frac{CR}{T} = log\frac{R}{nh} + \frac{\Delta S}{2.303R} - \frac{\Delta H}{2.303RT} \qquad (6)$$

Where CR is the corrosion rate at temperature T, R is the molar gas constant, n is Avogadro's constant, and h is the Planck's constant. A plot of $\log\frac{CR}{T}$ vs $\frac{1}{T}$ is a straight line graph from which the enthalpy and entropy change was obtained from the slope ($-\Delta H/2.303R$) and intercept ($\log (R/nh) + \Delta S/2.303R$) of the graph respectively. The values of activation energy (Ea), enthalpy (ΔH) and entropy (ΔS) change obtained for methanol, ethanol, aqueous and diethylether extracts are tabulated in table 3a and b respectively. The value of Ea in blank solution under methanol and ethanol extract were 7.16 kJ/mol while that of the aqueous and diethylether extract were 7.33 kJ/mol, and the values increase as concentration of inhibitors increases from 0.2g/L to 1.0g/L for all the solvents extracts. A physical adsorption mechanism is proposed for this study since the values of Ea are lower than 80 kJ mol^{-1} [17]. The increase in activation energy of the inhibited acidic solution indicates the deactivation of the acid molecules on collision with the metal surface by the introduction of the extract thus reducing the rate of acid attack on the metals. The manner of increasing in Ea values with increase in concentration of the extracts have been reported by earlier studies on various plant such as jujube leaves [18], black pepper [19], sunflower leaves [20], banana peels [21] and Centella asiatica leaves [22]. The enthalpy (ΔH°) values calculated for all solvents extracts are positive and it increases with increase in concentration of the inhibitor. The positive values of the enthalpy change indicated that the adsorption of the solvents extracts of the air dried alchornea laxiflora leaves on mild steel surface exhibited endothermic reaction while the increase in the values of enthalpy change with increase in concentration indicate that the addition of inhibitors retard the corrosion process and more energy is needed for it to break the film barrier and react with mild steel surface. Moreso the values obtained for the entropy (ΔS) change for all the solvents extracts of air dried alchornea laxiflora leaves are negatives, which indicates that there is a higher association of the extracts molecules rather than dissociation [23].



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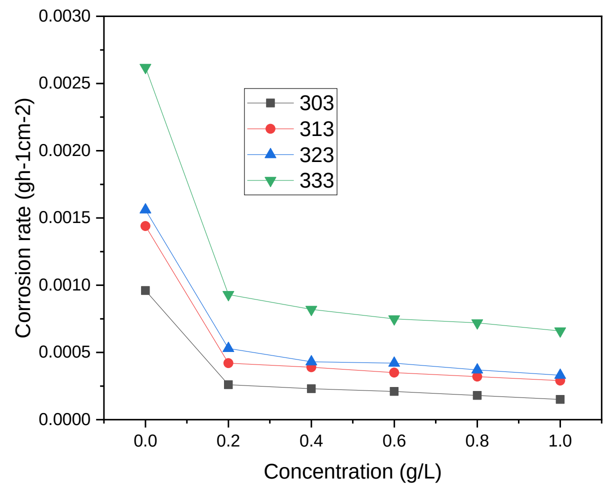

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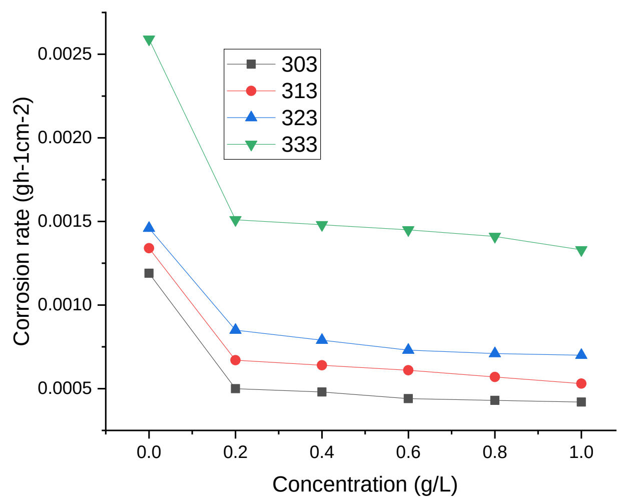

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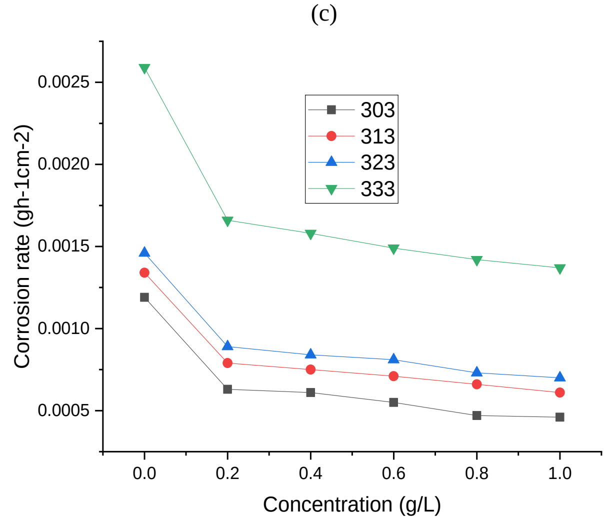
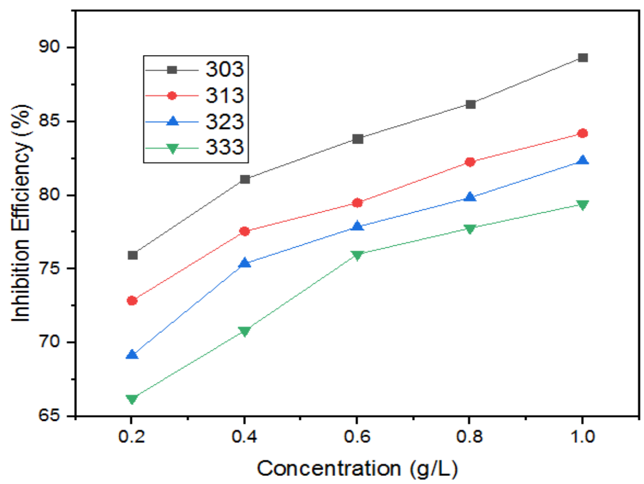


(d)

Figure 1: plot of corrosion rate of mild steel against Concentration of (a) Methanol extract (b) ethanol extract (c) aqueous extract (d) diethylether extract at different Temperature



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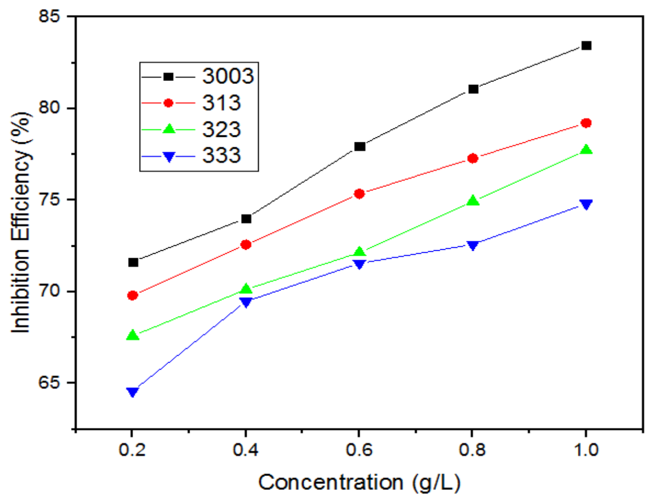

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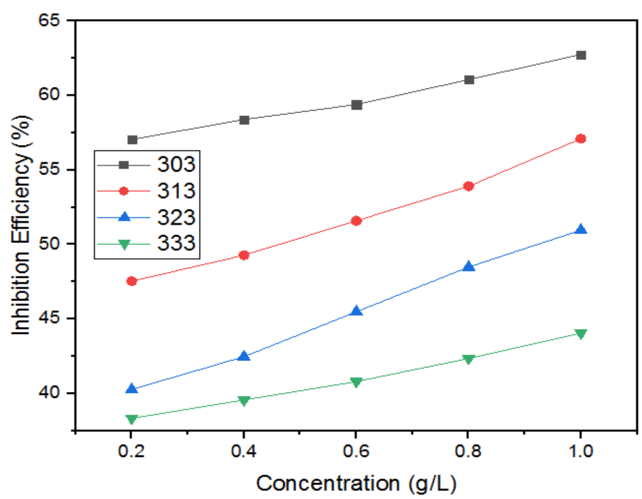

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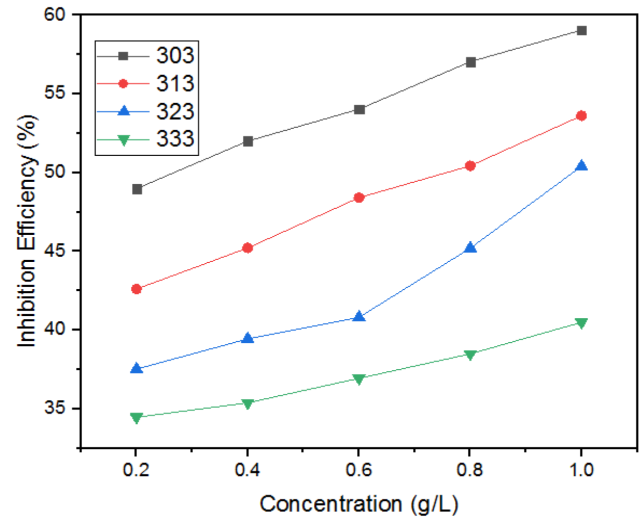


(d)

Figure 2: Plot of inhibition efficiency against Concentration of (a) Methanol extract (b) Ethanol extract (c) Aqueous extract (d) Diethylether extract at different Temperature.

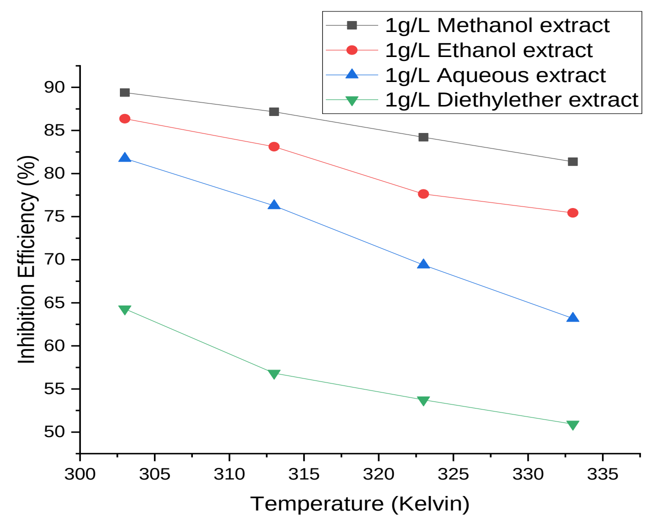


Figure 3: Plot of inhibition efficiency of the solvents extracts against Temperature at fixed concentration

Table 3a: Values of Ea, ΔHo and ΔSo in the absence and presence of methanol and ethanol extracts of air dried alchornea laxiflora leaves at different concentrations.

Conc. (g/L)	Methanolic extract			Ethanolic extract		
	Ea ($Kjmol^{-1}$)	ΔH ($Kjmol^{-1}$)	ΔS ($Kjmol^{-1}$)	Ea ($Kjmol^{-1}$)	ΔH ($Kjmol^{-1}$)	ΔS ($Kjmol^{-1}$)
Blank	7.16	4.53	-12.6721	7.16	4.53	-12.6721
0.2	12.30	9.68	-11.7001	13.72	11.09	-12.0651
0.4	14.33	10.70	-12.0981	14.71	13.09	-12.1371
0.6	14.49	11.55	-12.7121	19.82	17.17	-11.1871
0.8	14.50	11.87	-12.2401	23.70	21.08	-10.6041
1.0	18.62	15.98	-11.6651	27.02	24.39	-10.1451

Table 3b: Values of Ea, ΔHº and ΔSº in the absence and presence of Aqueous and Diethylether extracts of air dried alchornea laxiflora leaves at different concentration

Conc. (g/L)	Aqueous extract			Diethylether extract		
	Ea ($Kjmol^{-1}$)	ΔH ($Kjmol^{-1}$)	ΔS ($Kjmol^{-1}$)	Ea ($Kjmol^{-1}$)	ΔH ($Kjmol^{-1}$)	ΔS($Kjmol^{-1}$)
Blank	7.33	4.70	-12.6571	7.33	4.70	-12.6571
0.2	12.37	9.73	-12.1851	7.53	4.89	-12.8721
0.4	12.71	11.08	-12.3201	8.77	6.14	-12.6961
0.6	13.97	11.34	-11.9981	9.66	7.02	-12.7491
0.8	18.31	15.69	-11.3671	13.03	10.40	-12.0731
1.0	25.93	23.30	-10.1921	14.57	11.93	-11.8531

3.6 Adsorption Isotherm

The mechanism of action of most organic inhibitors in acidic medium is by adsorption and their mode of interaction could better be understood by using adsorption isotherms. Inhibition efficiency (IE) is directly proportional to the fraction of the surface covered by the adsorbed molecules (θ). Therefore, with the extract concentration specifies the adsorption isotherm that describes the system and gives the relationship between the coverage of an interface with the adsorbed species and the concentration of species in solution [24], [25]. The Values of the degree of surface coverage (θ) were evaluated at different concentrations of the different solvents extracts in 1.0 M HCl solution and were fitted to various adsorption isotherms. Different adsorption isotherms were tested in order to obtain more information about the interaction between the inhibitors and the mild steel surface. The various isotherms tested includes Temkin, Freundlich and Langmuir adsorption isotherms and the values obtained for all the solvents extracts are presented in Table 4a and b. The linear regression coefficients (r^2) were used to determine the best fit. Langmuir adsorption isotherms were found to be best fit in which case all the linear regression coefficients (r^2) were close to unity as shown on the Tables.

Table 4a: Adsorption isotherm parameters obtained from the corrosion data for mild steel in 1.0 M HCl containing (i) methanol extract and (ii) ethanol extract of air dried alchornea laxiflora leaves

(i)

	Langmuir adsorption			Freundlich adsorption			Temkin adsorption		
Temp.(K)	Slope	r2	K_{ads}	r2	1/n	Kf	r2	b_T [J/mol K]	A_T [L/g]
303	1.058	0.997	13.70	0.958	0.111	0.8851	0.951	64.46	18.2300
313	1.095	0.995	13.16	0.863	0.096	0.8492	0.851	77.83	60.6406
323	1.131	0.997	13.70	0.966	0.104	0.8318	0.958	77.31	31.6647
333	1.161	0.998	12.99	0.975	0.110	0.8110	0.969	77.76	19.0373

(ii)

	Langmuir adsorption			Freundlich adsorption			Temkin adsorption		
Temp. (K)	Slope	r2	Kads	r2	1/n	K_f	r2	b_T [J/mol K]	A_T [L/g]
303	1.069	0.996	9.71	0.942	0.145	0.8551	0.934	51.80	1.9947
313	1.133	0.996	10.87	0.966	0.125	0.8166	0.954	64.44	6.3948
323	1.232	0.998	13.33	0.970	0.099	0.7656	0.961	88.35	54.953
333	1.267	0.996	11.91	0.938	0.102	0.7396	0.926	91.09	37.925

Table 4b: Adsorption isotherm parameters obtained from the corrosion data for mild steel in 1.0 M HCl containing (i) Aqueous extract and (ii) Diethylether extract of air dried alchornea laxiflora leaves.

(i)

	Langmuir adsorption			Freundlich adsorption			Temkin adsorption		
Temp (K)	Slope	r2	Kads	r2	1/n	K_f	r2	b_T [J/mol K]	A_T [L/g]
303	1.087	0.971	5.29	0.836	0.202	0.7727	0.813	42.04	0.2689
313	1.200	0.971	5.38	0.826	0.182	0.7129	0.793	51.66	0.4629
323	1.317	0.984	5.68	0.874	0.169	0.6637	0.851	61.23	0.7091
333	1.446	0.994	6.62	0.941	0.156	0.6266	0.928	73.29	1.3184

(ii)

	Langmuir adsorption			Freundlich adsorption			Temkin adsorption		
Temp (K)	Slope	r2	Kads	r2	1/n	K_f	r2	b_T [J/mol K]	A_T [L/g]
303	1.382	0.980	4.33	0.887	0.207	0.6152	0.867	52.27	0.2526
313	1.634	0.986	5.21	0.863	0.152	0.5433	0.846	79.91	1.3760
323	1.738	0.992	7.87	0.956	0.151	0.5224	0.938	88.35	1.7076
333	1.851	0.994	6.02	0.945	0.134	0.4966	0.929	106.27	3.7653

3.7 Effect of immersion time

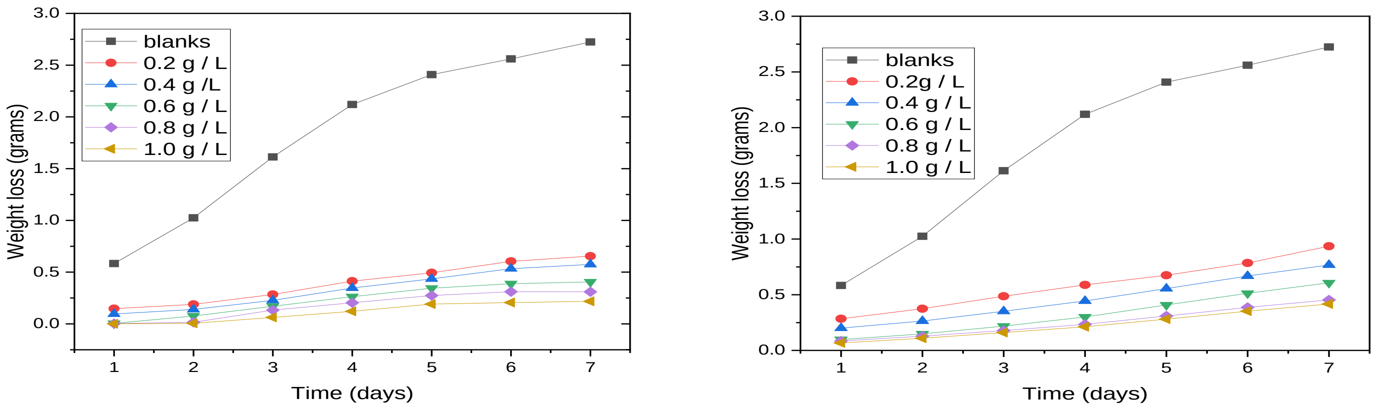
The effect of immersion time on the Weight loss of the mild steel in 1.0 M HCl was performed in the absence and presence of all the solvents extracts concentration (0.2 - 1.0g) for 7 days at room temperature. The plot of weight loss against time for the mild steel in the solvents extracts (Figure 4a and b) showed that weight loss of the mild steel increases as the immersion time increases in the blank and the solvents extracts solution but the weight loss is much more retarded in the solution containing the methanol extract of the plant leave. The reduction in the weight loss of the mild steel in the solution containing the methanol extract may be ascribed to its high percentage yield and phytochemical constituents which are readily available for adsorption on the surface of the mild steel.

3.8 Kinetic Study

In the kinetic study, the initial weight of mild steel coupon at time t, is designated as Wi, the weight loss is W_L and the weight change at time t, ($Wi - W_L$) while k, is the first order rate constant.

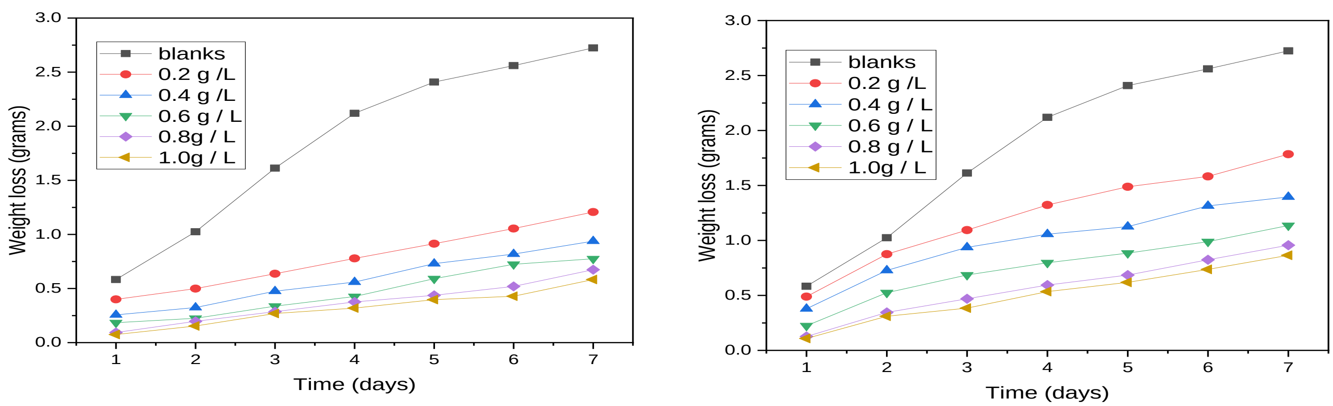
$$\ln (Wi - W_L) = -k_1 t + \ln W_L \quad (7)$$

According to equation 7, the plots of $\ln (W_i - W_L)$ against time (days) at room temperature showed a linear variation and the first order reaction rate constants (k) calculated from the slope of the graph and the half-life ($t_{1/2}$) for all the solvents extract were presented in Table 5.As observed from the Table 5, there is an increase in the half-lives ($t_{1/2}$) of the metals immersed in the 1.0M HCl containing the different extracts with methanol extract having the highest values. These work also aligned with our findings that the type of solvent used during extraction of the plant leaves contributes to its efficiency in corrosion prevention. It should also be noted that as the concentration of the extract increases, the half-life of the metal also increases, indicating decrease in the dissolution rate of the metal in the solution with increase in concentration of the inhibitor



(a) (b)

Figure 4a: Variation of weight loss with respect to time for corrosion of mild steel in 1.0 M HCl in the absence (blank) and presence of (a) Methanol extract (b) ethanolic extract extract of air-dried alchornea laxiflora leaves at different concentration



(a) (b)

Figure 4b: Variation of weight loss with respect to time for corrosion of mild steel in 1.0 M HCl in the absence (blank) and presence of (a) aqueous extract (b) diethylether extract of air-dried alchornea laxiflora leaves at different concentration

Table 5: Half-life and Rate Constants parameters at various concentrations of the different solvents extracts of air dried Alchornea laxiflora leaves

	Rate constants				Half life			
Conc.(g/L)	ME	EE	AE	DE	ME	EE	AE	DE
Blanks	0.0276	0.0276	0.1958	0.1958	25.1	25.1	3.5	3.5
0.2	0.0138	0.0230	0.0921	0.0967	50.2	30.1	7.5	7.2
0.4	0.0092	0.0161	0.0875	0.0898	75.3	43.0	7.9	7.7
0.6	0.0069	0.0115	0.0829	0.0875	100.4	60.3	8.4	7.9
0.8	0.0046	0.0092	0.0760	0.0806	150.7	75.3	9.1	8.6
1.0	0.0023	0.0046	0.0576	0.0645	301.3	150.7	12.0	10.8

4. Conclusion

The results of this work revealed that the type of solvent used during extraction of plant leaves is very critical in determine its percentage yield and the phytochemical constituents of the plant. The methanol extract of the air dried alchornea laxiflora leaves had the highest percentage yield and phytochemicals compositions compared to the ethanol, aqueous and diethylether extracts of the plant leaves. Though the inhibition efficiency of all the extracts increases as their concentration increases but decreases with increase in temperature with methanol extract having the highest inhibition efficiency. The thermodynamic parameters obtained from the study showed that the adsorption of the plant extracts on the surface of the mild steel is spontaneous and is in line with the physical adsorption mechanism. The parameters obtained in the kinetic study revealed that the half-life of the mild steel increased with increase in the concentration of the extracts with the methanol extract of the plant leaves having the highest half-life. Moreso, the adsorption of the leaves extract on mild steel follows Langmuir adsorption.

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